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Aug 1981

6 FRACTO-EMISSION FROM POLYMERS

10 J. Thomas/Dickinson

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number). Progress in the investigation of fracto-emission (FE) from polymers, composites, and other materials is summarized in this report. The unique emission from interfacial failure is described for fiber reinforced epoxy and polybutadiene filled with glass beads. Initial results of the determination of the mass of the positive ions, measurements of emission from Kevlar-epoxy, and a simple model for the observed decay from interfacial failure are discussed. Preliminary work including the role of cross-linking and the dependence of FE on crack velocity are mentioned.		

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I. Introduction

The major goals of the work this first year has been to examine the extent of fracto-emission phenomena accompanying and following the fracture of polymers, to measure the properties of this emission, and to determine the physical and chemical parameters which influence fracto-emission. A Technical Report describing some of this year's progress has recently been published, namely: Technical Report No. 1, "The Emission of Electrons and Positive Ions from Fracture of Materials," by J. T. Dickinson, E. E. Donaldson, and M. K. Park.¹ Only a summary of this work will be presented here. Recent extensions and new results will be noted also.

II. Results and Discussion

A. Survey of Fracto-Emitters

A wide range of materials were fractured in vacuum and the resulting charged particle emission detected. Detectors for both negative and positive charge emission were used. All materials were strained to failure, usually in tension. For brittle materials, three-point bending strain was used. Materials ranged from crystalline inorganic salts, ceramics, sugar, glassy polymers, elastomers, to composites. In all materials tested, emission was observed. The intensity and time dependence of the emission was material dependent.

For a few materials we used magnetic fields to probe the negative charge emitted for a negative ion component. No significant negative ion component was observed; i.e., the negative particles we observe appear to be predominantly electrons.

The intensity of the emission per unit cross-sectional area goes up as sample cross-section decreases. This may be due to either a higher probability of escape of the particle or the increase in elastic strain energy stored in the material (filaments have higher tensile strength).

B. Interfacial Failure in Composites

In systems where polymer/inorganic solid interfaces fail, we observe extremely intense and long lasting emission; millions of charged particles with emission that lasts for several tens of minutes. Much lower emission lasting only microseconds is observed when the separate constituents of the composites (e.g. glass, graphite, Kevlar, and epoxy) are fractured. Accompanying interfacial failure is considerably more charging of the surfaces

perhaps due to both emission and charge separation. In air, these surfaces are above the break-down potential of air, suggesting that the resulting surface charge density is not at all at its maximum. We have evidence that the density of surface charge is playing an important role in our observed FE. For example, the energy distributions of the electron and emission (EE) positive ion emission (PIE) are nearly identical and have a peak near 0 eV and a slowly decreasing tail that extends beyond 2 keV, the limit of our retarding potential energy analyzer. There have been reports of 40 keV electrons from interfacial failure between teflon and epoxy² and 120 keV electrons from cleavage of alkali halides.³ Our interpretation of such measurements is that intense charging due to charge separation produces the electric fields that accelerate the emitted charges to these high energies.

In terms of phenomena accompanying crack growth in systems at high pressures, this charge separation could lead to considerable energy release in the form of breakdown and corona discharge as well as additional emission and surface bombardment by charged particles due to cascade effects.

C. Interfacial Failure in Filled Elastomers

When polybutadiene and solithane filled with glass beads is fractured we observe analogous effects like the composites: long, intense emission with a very similar decay curve lasting for several minutes. The emission intensity is sensitive to the volume percent of glass beads, increasing with bead concentration. Again, this enhanced emission is attributed to interfacial failure at the surface of the beads. The energy distributions of the EE and PIE show energies that fall from near zero eV to energies above 2 kV. Again, the role of intense charging could be significant at high pressures. The crack-tip would be filled with volatiles produced

by the fracture itself. Breakdown, corona discharge, and/or fracto-emission interacting with the gases and crack-walls could result in a number of chemical effects.

III. Most Recent Results

A. Mass of Positive Ions

Several attempts to measure the mass of the PIE emitted from polymer fracture were made using a quadrupole mass spectrometer. Because of the broad energy distribution extending to high energies, the quadrupole could not mass discriminate. Ions simply passed down the quadrupole section undeflected.

We have made progress in separating out the lower part of the energy distribution with a grid system and introducing bursts of these ions into a time-of-flight (TOF) mass spectrometer. With relatively low resolution we have examined the PIE from delamination of 3M Filament Tape; i.e. the separation between the polyester backing and the filaments which adhere by means of a natural rubber based adhesive. This mode of failure produces intense EE, PIE, as well as photon emission. The mass was determined by measuring the shift in the TOF spectrum as a function of voltage applied to the drift tube which was 25 cm in length. The crude value obtained was 120 ± 60 amu. Although the uncertainty is high, the measurement indicates that the PIE consists of relatively small fragments, possibly monomers or chain fragments produced by bond scission. We are attempting to improve our mass resolution and ion optics to better determine the PIE masses.

B. Kevlar and Kevlar-Epoxy Fracture

Similar to measurements performed on glass- and graphite-epoxy systems we also examined the EE and PIE from the fracture of 10μ Kevlar filaments DER 332, similar to the strands studied earlier. Figure 1 shows the PIE time distribution for the Kevlar filaments which decays exponentially with a time constant of $45 \mu s$, similar to a number of polymers and certainly longer than

PIE TIME DISTRIBUTION FOR KEVLAR FILAMENTS

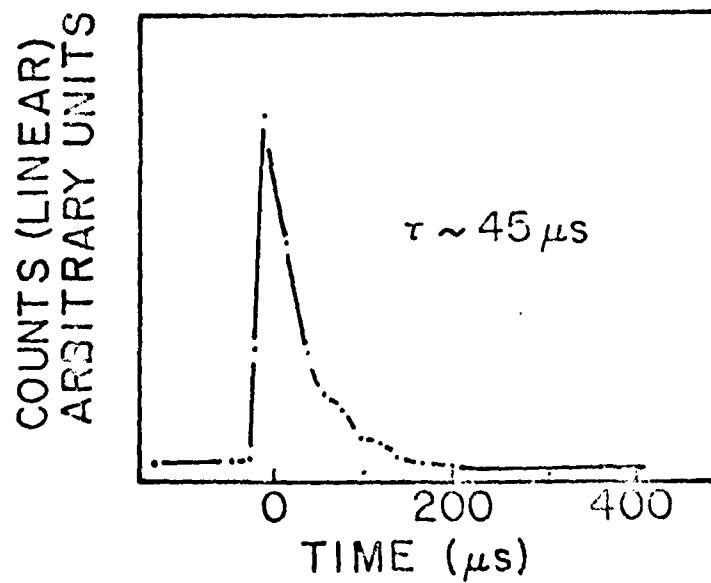


Figure 1. Time distribution for positive ion emission due to fracture of 10 micron Kevlar filaments. The time constant for the exponential decay is about 45 microseconds.

EE AND PIE FROM
KEVLAR/EPOXY STRANDS

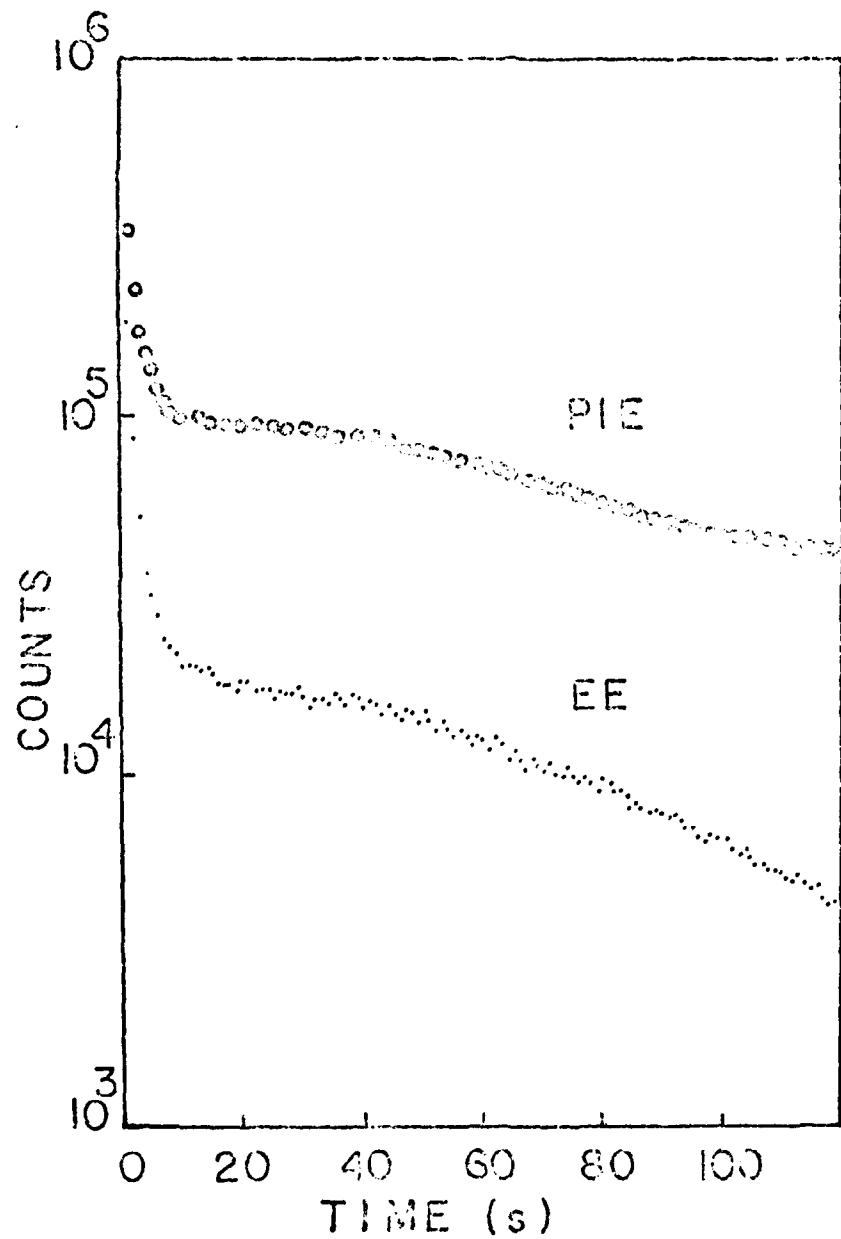


Figure 2. EE and PIE from the fracture of Kevlar/Epoxy strands on a log plot. The 10 micron kevlar filaments were in a DER 332 epoxy.

EE AND PIE ENERGY DISTRIBUTIONS
FROM KEVLAR-EPOXY STRANDS

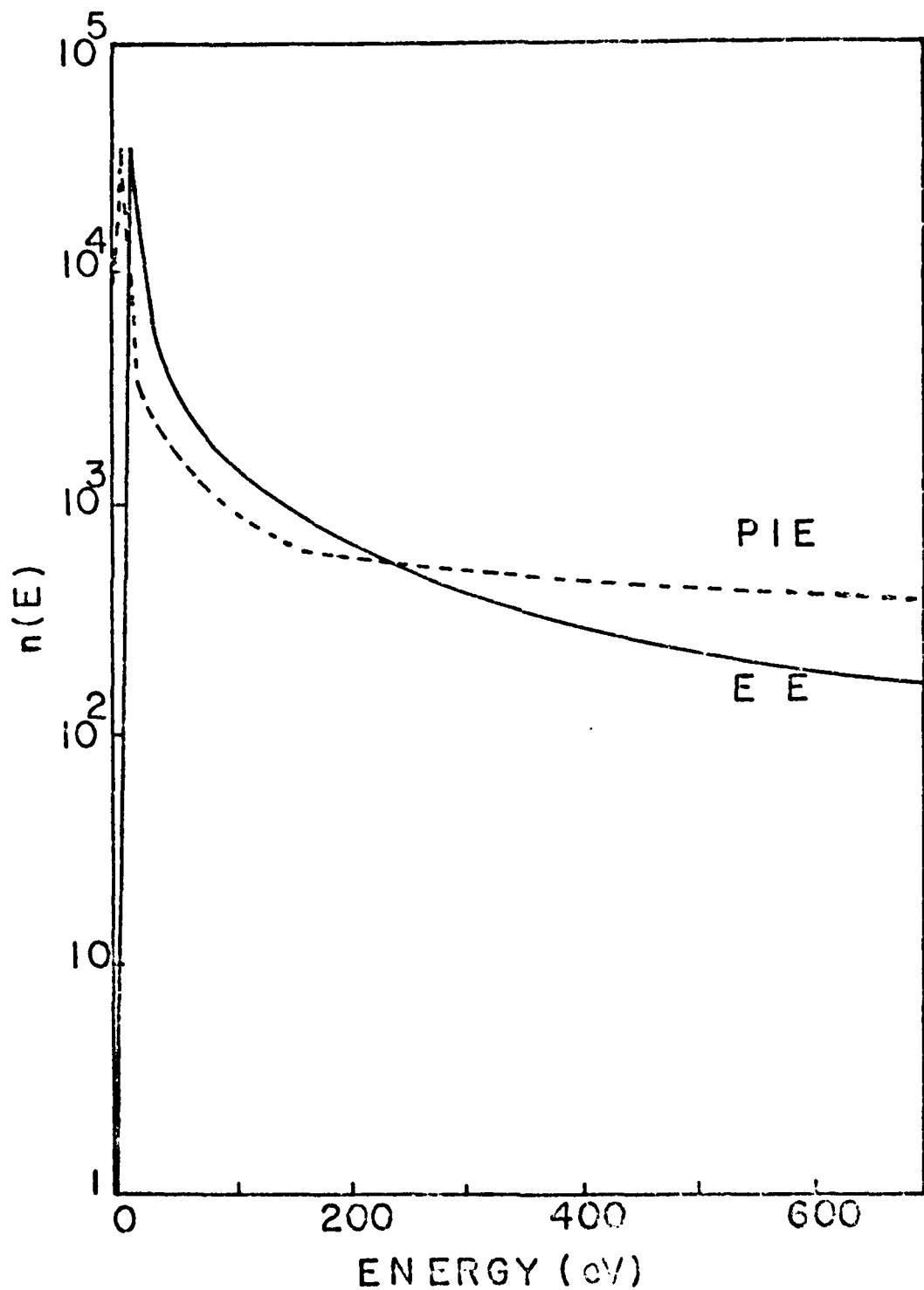


Figure 3. Energy Distributions for the EE and PIE due to fracture of Kevlar-Epoxy strands over the range 0-600 eV.

the inorganic filaments which had decay times of about 10 μ s. The EE time distributions are quite similar.

If we then examine EE and PIE from Kevlar/Epoxy as seen in Fig. 2, we see that the emission is very long lasting. It has the characteristic initial decay from the peak, but has a departure in slope from simple exponential decay. This has been seen on a number of other systems involving adhesive failure (unpublished) and following the cleavage of mica. Nevertheless, the intense, long lasting emission again is attributed to the enhanced excitation that occurs from interfacial failure between the Kevlar filaments and epoxy. The intensity is higher than other filament-epoxy systems studied and so is the extent of interfacial failure. Fig. 3 shows the energy distributions on a log plot for both the EE and PIE.

C. Computer Fit of EE from E-Glass/Epoxy Strands

The decay from the composite systems has the appearance of either two exponential terms (suggesting two independent first order mechanisms at constant temperature, or a modified first order process that decays to a simple first order curve. Figure 4, curve A is a nonlinear least-squares fit of a typical EE decay curve for E-glass/epoxy using two exponential terms. The best fit was obtained with time constants 0.83 s and 21 s. C_1 and C_2 were also adjustable parameters. Although the fit looks fairly good, one sees that there are deviations.

An improved fit of the data was obtained by assuming a simple first-order process given by:

$$\frac{dN}{dt} = v N \exp(-E_A/kT)$$

where N = the number of excitations whose decay produces electrons, v = a pre-exponential factor, E_A = activation energy for thermal stimulation of the excitation decay. We also assume a rise in temperature at the surface where the sites

EE FROM E-GLASS EPOXY STRAND

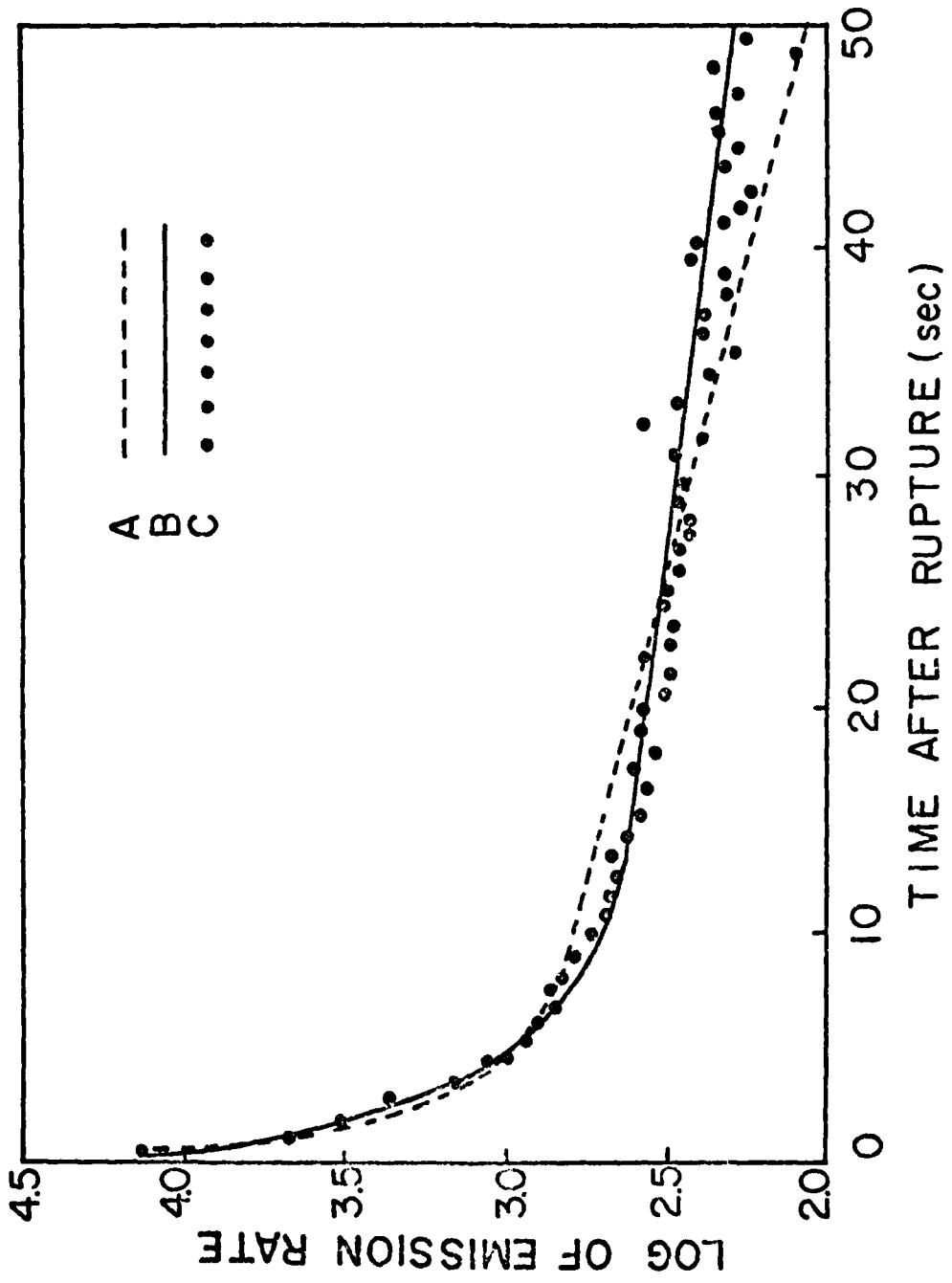


Figure 4. Log plot of the first 50 seconds of the EE from E-Glass/Epoxy. The dots are the data points. Curve A (dashed line) represents a non-linear least squares fit to the equation $C_1 \exp(-t/\tau_1) + C_2 \exp(-t/\tau_2)$; where $\tau_1 = 0.83$ s and $\tau_2 = 21$ s. Curve B (solid line) represents the least squares fit to the first order differential equation with a rise in temperature that falls exponentially.

exist. This temperature is expected to fall exponentially as follows:

$$T(t) = T_{\text{room}} + \Delta T(\exp - t/\tau) .$$

Figure 4, curve B shows the fit obtained with the parameters $\Delta T = 400^\circ\text{C}$, $E_A = 0.1 \text{ eV}$, and $\tau = 2.6 \text{ s}$.

This model is being applied to other data sets for filled polymers and other epoxy-filament systems. If valid, it suggests a way to determine the surface temperature immediately after fracture. It would also predict that at elevated temperatures the rate of decay of the after-emission would be higher, which we plan to check experimentally.

D. Other Current Work

Other work which is too preliminary to report here will just be mentioned for completion.

1) We have measured the EE and PIE from un-notched polybutadiene filled with glass beads under tensile strain before rupture. We see a clear peak in emission that appears when the material "whitened" presumably due to detachment of the beads from the polymer matrix. The particle emission we observe is most likely due to this interfacial failure where the vacuola created opens a portion to the vacuum. The latter is necessary for the released particles to reach the detector. Measurements are being made of the strain at onset of this emission to indicate when bead detachment is initiated. Also, the shape of the emission vs. strain curve may be related to the distribution of detachment vs. strain.

2) We have measured the total emission and decay curves of unfilled elastomers, namely SBR (butadiene-styrene co-polymer) and polybutadiene before and after exposure to UV-radiation or γ -rays (3000 Curie Co-60 source). Both types of radiation produced enhanced emission both

during and following crack propagation. Swelling measurements show that the degree of cross-linking increased. It is felt that because of enhanced cross-linking, fracture produces more molecular fragments and free-radicals that can enter into the emission process. This work is being quantified.

3) Crack-tip motion in elastomers at room temperature is sufficiently slow that elementary methods can be used to measure displacement vs. time while simultaneously measuring EE or PIE. We are using a standard video camera and recorder. Our recent measurements show a strong dependence of FE intensity on crack velocity. In some materials we have found that on millisecond time scales, our detectors are completely saturated (implying detector count rates $\geq 10^6$ per second) at crack velocities $\approx 10^2$ cm/sec. In order to see the shape of the emission curves vs. time at high velocities we plan to replace the detector with a standard electron multiplier which has a lower sensitivity and a larger dynamic range. At some point we will want to increase our strain rate and crack-motion measurements.

IV. Conclusion

The underlying significance of our results to date on FE from polymers is that when a crack propagates through the material the fracture surfaces are in a highly excited, nonequilibrium state. This involves broken bonds, liberated fragments (e.g. free radicals, atoms, charged species), defects, intense charge separation (particularly in the case of interfacial failure), and a localized rise in temperature. The observed FE is a measure of the degree of this excitation and is evidence of considerable chemical activity. The fracture event produces activated species, such as free radicals, that undergo recombination reactions releasing sufficient energy to produce particle and photon emission. The charging of surfaces certainly is responsible for the high energy particles observed and may also effect the emission intensity. This could be due to an influence on the mobility of active species near and on the fracture surface as well as a direct coupling of the E-field to electronic transition probabilities in excited species. The fact that we see such enhanced effects when interfaces are involved is likely due to a) the different types of chemical species produced by adhesive failure (as compared to cohesive failure) and b) the larger degree of charging that seems to occur with interfacial failure.

In this report we have outlined the progress of our first year's efforts to study the Fracto-Emission from polymers. We have presented evidence of such emission for a wide range of materials and shown that interfacial failure produces intense, long-lasting emission. We are finding that the emission is probably involving the production and decay of free radicals and that surface charging is playing an important role, perhaps in the filling of newly created surface states and influencing the mobility of active

species. The potential of using FE for detecting filler detachment, measuring crack-tip motion at very high crack velocities, and relating crack motion to polymer morphology appears very high. Also, FE can serve as a probe on the microsecond time scale of the activity of the freshly created crack walls. All these studies have direct applicability to fracture phenomena in energetic materials.

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M. K. Park, Graduate Student	50%
D. B. Snyder, Graduate Student	10%
B. J. Tillotson, Undergraduate Student	10%

VII. Fracto-Emission Talks and Papers Presented

"Electron and Acoustic Emission Accompanying Oxide Coating Fracture," International Conference on Metallurgical Coatings, San Diego (1980).

"Electron and Positive Ion Emission During Fracture of Oxide Coatings," AVS Conference, Detroit (November, 1980).

"Fractoemission," WSU, Department of Materials Science (December, 1980).

"Charged Particle Emission Accompanying Fracture," Materials Science Branch, NASA-Ames, December, 1980).

"Electron and Positive Ion Emission Accompanying Fracture," Navy Surface Weapons Center (October, 1980).

"Fractoemission from Polymers," Third ONR Meeting on Dynamic Fracture of Elastomers (February, 1981).

"Fractoemission from Solids," Lawrence Livermore Laboratories (March, 1981).

"Exoelectron Effects During Fracture," Conference on Stress Corrosion in Brittle Materials, NBS (June, 1981).

"Fractoemission from Composites," DOD-TTCP Critical Review: Characterization of Composite Materials, MIT (June, 1981).

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